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FED TEST METHOD STD. 191A
CHANGE NOTICE 4
March 31, 1987

FEDERAL TEST METHOD STANDARD

TEXTILE TEST METHODS

The following changes, which form a part of FED TEST METHOD STD. 191A, dated July 20, 1978, are approved by the Assistant Administrator, Office of Federal Supply and Services, General Services Administration for the use of all Federal agencies.

ADD : Tentative Standard Test Method 2017.

REMOVE : Standard Test Method 2050 of July 20, 1978.

ADD : Revised Standard Test Method 2050.1.

REMOVE : Standard Test Method 5905 of July 20, 1978.

ADD : Revised Standard Test Method 5905.1.

MILITARY INTEREST:

Custodians

Army - GL
Navy - NU
Air Force - 20

Review Activities

Army - AR, EA, MD, ME, TE
Navy - AS, SH
Air Force - 11, 82, 99

CIVIL AGENCY COORDINATING ACTIVITIES:

GSA - FSS
HHS - NIH

PREPARING ACTIVITY

Army - GL

Project No. 83GP-0029

RETAIN THIS CHANGE NOTICE AND INSERT BEFORE THE TABLE OF CONTENTS

FSC 83GP

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METHOD 2017
March 31, 1987

SODIUM SALT CONTENT OF [(4,5-DICHLORO, 2-CHLOROMETHANE SULFONAMIDE) 3', 4', 6'
TRICHLORO] DIPHENYL ETHER

1. SCOPE

1.1 This method is intended to determine the sodium salt content of [(4,5-Dichloro, 2-Chloromethane Sulfonamide) 3', 4', 6' Trichloro] diphenyl ether content of woolen textile materials that have been treated with this compound as a mothproofing agent (see 7.1).

2. TEST SPECIMEN

2.1 All wool. When the material to be tested is 100 percent wool, the specimen shall be 5.0 ± 0.20 grams.

2.1 Polyester and wool blend. When the material to be tested is a blend of polyester and wool, the specimen shall be 10.00 ± 0.50 grams.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the procurement document, two specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Electric heater with variable control.

4.1.2 Soxhlet extraction unit with 300 mL glass flask.

4.1.3 100 mL volumetric flask.

4.1.4 250 mL volumetric flask.

4.1.5 Gas chromatography with electron capture detector, with (if available) linearized indication, recorder and integrator; with 6 foot column filled with chromosorb w(80/100 mesh) with 3 percent silicon rubber SE-30 (see 7.2).

4.1.6 5 or 10 microliter syringe.

4.1.7 Analytical balance.

4.1.8 Laboratory drying oven.

METHOD 2017

4.1.9 Desiccator.4.2 Reagents.

4.2.1 2-methoxy ethanol (methylglycol). Free of electron affine impurities.

4.2.2 95 percent Argon/5 percent methane mixture as carrier gas.

4.2.3 20 mg/1 solution of [4, 5-dichloro, 2-chloromethane sulfonamide) 3', 4', 6' trichloro]] diphenyl ether (see 7.3). Weight $5.0 \pm .1$ milligram of the 100 percent active material, dissolve in a 2-methoxy ethanol and fill to 250 mL volume in a volumetric flask (see 7.4).

5. PROCEDURE

5.1 Weight of dry specimen. The specimen shall be placed in a weighing bottle, dried in a circulating air oven at a temperature of 105°C to 110°C, cooled in a dessicator, and weighed. Repeat this cycle until a weight that is constant to ± 0.001 gram is obtained. This is the "weight of the dry specimen" and in the calculation of results is indicated as "W".

5.2 Testing of specimens fabricated from 100 percent wool or of polyester/wool blend. Cut specimen into small pieces and place in a soxhlet extractor. Extract for 10 cycles (approx. 2 hours) using 2-methoxy ethanol as a solvent (see 7.5). After cooling, transfer quantitatively into a 100 mL volumetric flask and fill to volume with 2-methoxy ethanol.

Adjust the gas chromatography to the following conditions:

oven temperature - 240°C isothermal
injection temperature - 250°C
detector temperature - 300°C
run time - 10 minutes
flow rate - 50 mL/min
chart speed - 1 cm/min
attenuation - 2 9
slope sensitivity - 200
area rejection - 10

Inject exactly 1 microliter of the standard solution and 1 microliter of the extract of unknown content. Repeat the injections one time each. Record the adsorption peaks obtained (see figure 2017).

5.3 Calculations.

5.3.1 Integrate the 7-10 absorption peaks obtained with a suitable technique to determine the sum of the areas under the peaks, and normalize the sum, if necessary, to equal sensitivity adjustment. Determine the average of the sums of the two tests for the standard (Fs) and of the two tests for the unknown sample (Fp).

5.3.2 Specimens fabricated from 100 percent wool. The percent mothproofing agent on the wool fiber shall be calculated as follows:

$$\text{Percent mothproofing agent} = \frac{S \times F_p \times DF_p}{F_s \times W \times P} \times 100$$

Where:

S = parts per million of 100 percent active material dissolved in 250 mL 2-methoxy ethanol standard solution.

W = the original dry weight of the textile sample in grams (5.1).

DFp = unknown sample dilution factor which gives an average peak sum in the Linear portion of the Edolan U Standard curve.

Fp = the average peak sum of the unknown sample solution in relative units (5.3.1).

Fs = the average peak sum of the standard solution in relative units (5.3.1).

5.3.3 Specimens fabricated from polyester/wool blends. The percent mothproofing agent on the wool fiber in the blend shall be calculated as follows:

$$\text{Percent mothproofing agent} = \frac{S \times F_p \times DF_p}{F_s \times W \times P} \times 100$$

Where:

S, Fp, Fs and W have been defined under 5.3.2.

P = the proportion of wool in the sample, expressed as a decimal to the nearest 0.01.

6. REPORT

6.1 The percent mothproofing agent content of a sample unit shall be reported as the average of the values obtained for the specimens and shall be reported to the nearest 0.1 percent.

METHOD 2017

6.1.1 The individual values for each individual specimen used to calculate the average shall be reported to the nearest 0.01 percent.

7. NOTES

7.1 This method determines the content of mothproofing agent as 100 percent active (pure) material and not as commercial product. The commercial product has a concentration of active material of 33.3 ± 0.5 percent.

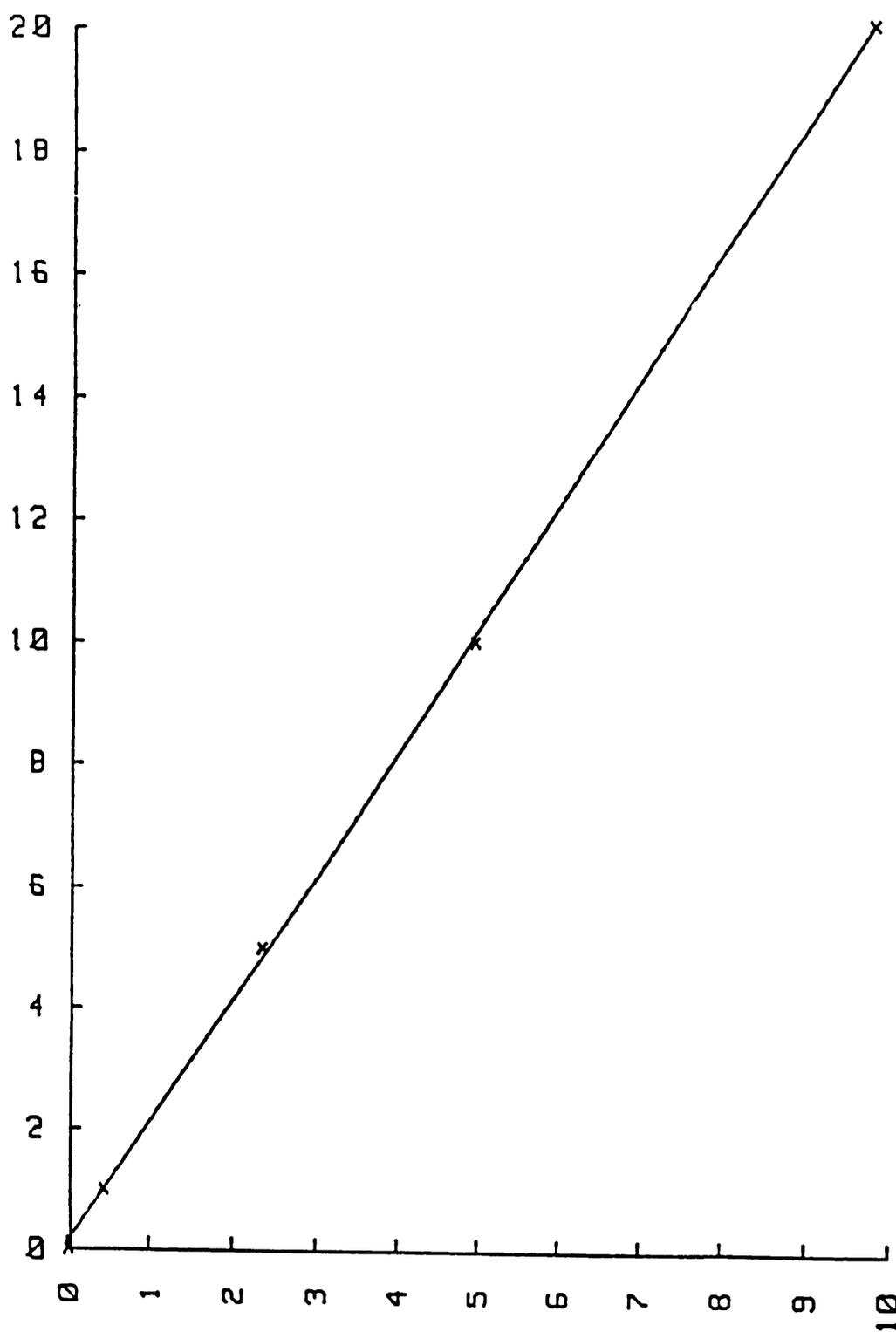
7.2 A Hewlett-Packard Series 5710A gas chromatography with electron capture detector is suitable for the determination. Other comparable equipment and column materials may be substituted. The suitability should be determined by making test runs with standard solutions of known concentrations.

7.3 One hundred percent of active material [(4, 5-dichloro, 2-chloromethane sulfonamide) 3', 4', 6' trichloro] diphenyl ether can be obtained from Dyes, Pigments and Organics Division of Mobay Corp., P. O. Drawer 2855, Rock Hill, SC 29730.

7.4 If the gas chromatography output is not linearized, the concentration of the standard solution shall be adjusted so that the sums of the peak areas for standard and sample are approximately the same or standard solution at several concentrations shall be used.

7.5 The described extraction method removes 99 percent or more of the active material on the fiber. Aside from the active mothproofing agent the 2-methoxy ethanol extract can contain other extractable substances such as dyes. These have not been found to have any influence on the determination of the active content of mothproofing agent.

EDOLAN U STANDARD CURVE



AREA ($\times 10^6$)

FIGURE 2017

COPPER CONTENT OF TEXTILES, ATOMIC ABSORPTION METHOD

1. SCOPE

1.1 This method is intended for determining the total copper content of textiles which have been treated with copper compounds. Presence of other metals in solution does not interfere with determination of the copper content.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the procurement document, the specimen shall be approximately 0.5 g of the material. (Specimen size can be increased or decreased dependent on the anticipated copper content of the material.) The material shall be well shredded or cut into small pieces prior to weighing the specimen.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the procurement document, two specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Analytical balance.

4.1.2 Weighing bottle.

4.1.3 Air oven.

4.1.4 Dessicator.

4.1.5 Kjeldahl flask.

4.1.6 Kjeldahl digestion apparatus (see 7.1).

4.1.7 Funnel.

4.1.8 Filter paper.

4.1.9 Volumetric flask.

4.1.10 Pipettes.

METHOD 2050.1

4.1.11 Atomic absorption spectrophotometer. Any commercial atomic absorption spectrophotometer that gives accurate results with solutions of known copper content may be used (see 7.2).

4.2 Reagents. All reagents shall meet ACS specifications.

4.2.1 Copper standard. (see 7.3).

4.2.2 Distilled water (copper free).

4.2.3 Nitric acid. (HNO_3) specific gravity 1.42.

4.2.4 Sulfuric acid. (H_2SO_4), specific gravity 1.84.

5. PROCEDURE

5.1 Unless otherwise specified in the procurement document, the specimen shall be placed in a tared weighing bottle, dried in an oven for 2 hours at a temperature of 221°F to 230°F (105° to 110°C), cooled to room temperature in a desiccator, weighed to the nearest 0.1 mg, and the dry weight of the specimen calculated.

5.2 Standard oxidation procedure.

5.2.1 The specimen from 5.1 shall be placed in a 30 mL Kjeldahl flask (see 7.4). Add 5 mL of 1:1 mixture (v/v) of concentrated sulfuric and nitric acids to the flask. Flask shall be placed on a Kjeldahl digestion apparatus and warmed. Kjeldahl digestion apparatus shall be either ducted for adequate ventilation or located in a fume hood. When initial reaction subsides, increase heat until specimen chars. Add a few drops of concentrated nitric acid cautiously and gradually increase heat. If specimen turns black, add more nitric acid and increase heat until specimen no longer chars. Digestion shall be continued with increased heat until white fumes are produced.

5.3 The digest shall be cooled and cautiously diluted with 10 to 15 ml of distilled water. Transfer the diluted digest to a 100 mL volumetric flask. Rinse down the sides of the Kjeldahl flask with distilled water and transfer the washing to the volumetric flask. Repeat the rinse procedure two more times and transfer all washings to the the volumetric flask. Fill volumetric flask to mark with distilled water and mix thoroughly by shaking. Filter the solution from the volumetric flask through a medium porosity qualitative filter paper.

5.4 Determination of copper content.

5.4.1 The atomic absorption spectrophotometer shall be set up in accordance with the recommendations of the manufacturer. The spectrophotometer shall be calibrated with a reagent blank and suitable copper standards (see 5.5). After calibration, the filtrate shall be aspirated into the spectrophotometer and each measurement recorded. Measurements should fall between the extremes of the

calibration limits. If not, appropriate measures shall be taken to either adjust spectrophotometer operating conditions or concentrate/dilute the filtrate until the filtrate is within the limits of the calibration range.

5.5 Calibration of spectrophotometer.

5.5.1. The spectrophotometer shall be calibrated in accordance with the recommendations for the spectrophotometer. For a working range up to 25 mg/L, the copper standards shall be prepared as follows: A copper stock solution (100 mg/L) shall be prepared by diluting 10 mL of copper standard (containing 1000 mg/L) to 100 mL in a volumetric flask. Fill volumetric flask with distilled water and shake thoroughly. Aliquots of 1 to 25 mL of the copper stock solution (100 mg/L) shall be pipetted into separate 30 mL Kjeldahl flasks for preparation of copper standards corresponding to 1 to 25 mg/L. Some of the standards shall be used as calibration standards and the rest shall be used as check standards to test the accuracy of calibration. One flask without copper standard shall be used for preparation of a reagent blank. The procedure from this point shall be as described in 5.2 through 5.3. The spectrophotometer shall be internally calibrated with calibration standards, and check standards shall be measured directly as mg/L.

5.6 Calculations.

5.6.1 Copper concentration of each filtrate shall be read directly from the spectrophotometer as mg/L. The copper content shall be based on the weight of the over-dried specimens and shall be calculated as follows:

$$\text{Copper, percent} = \frac{\text{Concentration of filtrate (mg/L)}}{\text{Weight of specimen (g)} \times 100}$$

6. REPORT

6.1. The percent of copper of the sample unit shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 0.01 percent.

6.1.1 The individual values for each individual specimen used to calculate the average shall also be reported.

7. NOTES

7.1 Rotary Kjeldahl digestion apparatus suitable for use in this method may be purchased from Kontes Scientific, Vineland, New Jersey 08360, or from another supplier (see 7.4).

7.2 Atomic absorption spectrophotometer suitable for use in this method may be purchased from Perkin-Elmer Corporation, Norwalk, Connecticut 06858, from Allied Analytical Systems, Andover, Massachusetts 01810, or from another supplier.

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7.3 Copper standard containing 1000 mg/L shall be prepared by dissolving 1.000 g of copper metal in a minimum volume of 1:1 (V/V) nitric acid and distilled water. Copper concentrate shall be diluted to 1 liter with 1% (v/v) nitric acid. Alternatively, copper standard containing 1000 mg/L may be purchased from Fisher Scientific Company, Pittsburgh, Pennsylvania 15219, or from another supplier.

7.4 Alternatively, the specimens from 5.1 may be digested in a 50 mL Purex beaker covered with a ribbed beaker cover (or another type of glassware equally suitable for digestion). Flat-bottomed glassware may be heated on a hot plate. Warning: Digestion in a flat-bottomed vessel may require more than 5 mL of 1:1 nitric and sulfuric acids to cover the vessel bottom.

FLAME RESISTANCE OF MATERIAL: HIGH HEAT FLUX CONTACT

1. SCOPE

1.1 This method is intended for use in determining the resistance of textiles and other materials to flame propagation.

1.2 This method provides a means to identify changes that occur in thermo-plastic materials on flame contact such as melting or shrinking from the flame source and is useful in predicting the flame resistance of materials which are not ignited by a low heat flux ignition source but from flammable decomposition products when subjected to a higher heat flux,

1.3 This method is intended to complement Method 5903 and provides a higher heat flux flame contact and a longer exposure time to flame source. The heat flux is comparable to that of the ignition source (6 pounds (2.7 kg) of paper strips set with a 3-foot (914 mm) length) used in a tent burning test.

2. TEST SPECIMEN

2.1 The specimen shall be a rectangle of material 2-3/4 inches by 12 inches (70 mm by 305 mm) with the long dimension parallel to either the warp or filling direction of the cloth. For woven materials, no two specimens shall contain the same warp and filling yarns.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the procurement document, five specimens from each of the warp and filling directions shall be tested from each sample unit.

4. APPARATUS

4.1 Burner. Fisher burner, high temperature, liquefied petroleum type (see 7.1.2).

4.2 Crucible tongs.

4.3 Support stand.

4.4 Utility clamp.

4.5 Stop watch. Stop watch or other device to measure the burning time to 0.2 second.

METHOD 5905.1

4.6 Butane gas C.P. (see 7.1.1).

4.7 Gas regulator valve system. A control valve system with a delivery rate designed to furnish gas to the burner under a pressure of $2\text{-}1/2 \pm 1/4$ pounds per square inch (175 ± 17.5 g/cm²) at the reducing valve. The flame height is adjusted at the reducing valve reducing a pressure at the burner of about 0.1 pounds per square inch (45 g/cm²) (see 7.1.1).

4.8 Measuring scale. Measuring scale or metal tape graduated in increments of 1/8 inch (1 mm) to measure the length of char.

5. PROCEDURE

5.1 The material undergoing test shall be evaluated for characteristics specified in the applicable procurement document, i.e., ignition resistant, self-extinguishing, after-flame time, non-melting, does not drop flaming pieces from the material, non-shrinking, and percent consumed.

5.2 All specimens tested shall be at moisture equilibrium under Standard Atmospheric Conditions in accordance with Section 4 of this Standard.

5.3 The ignition source is a high-temperature burner with butane gas fed through the control valve system. Adjust the air inlet valve of the burner to half open. Open the burner needle valve approximately 1/2 turn from the closed position. Turn on the gas. Adjust the total flame height to approximately 3 inches (76 mm) using a ruler or a metal spur attached to the burner to estimate the flame height.

5.4 Grasp the top edge of the specimen with the crucible tongs and suspend it vertically in the flame for 12 seconds. The specimen is held with the lower edge 1-1/2 inches (38 mm) above the center of the burner. If the specimen shrinks or changes in shape, it shall be moved so that the lowest part of the specimen is in the required position. At the end of the 12-second period, withdraw the specimen slowly. Time any after-flaming that occurs (see figure 5905A) .

5.5 For specimen which may block the burner flame with melt, the burner, after adjusting the flame as described in 5.3, is clamped to a stand in a horizontal position 6 inches (152 mm) above the stand base. The specimen is introduced into the flame in a vertical position and the test is carried out as described in 5.4.

5.6 Definitions.

5.6.1 Ignites, propagates flame. The test specimen ignites and is completely consumed by flame either during flame contact or after withdrawal from the burner; or, flaming melt from the test specimen propagates flame, i.e., melt flames as it leaves the test specimen.

5.6.2 Ignites, self-extinguishing. The test specimen flames during or after withdrawal from flame exposure but is self-extinguishing before the entire length of the specimen is consumed.

5.6.3 Ignition resistant. No flaming of the specimen occurs during exposure or after withdrawal from the flame source (see figure 5905B).

5.6.4 Melts. The portion of the specimen in contact with the flame turns to a viscous liquid and flows, separating from the specimen.

5.6.5 Shrinks. The specimen in contact with the flame withdraws from it by shrinking, or breaking up without forming a liquid melt (see figure 5905C).

5.6.6 Drops flaming pieces. A part of the specimen breaks away or melts away from the specimen and is flaming as it leaves the specimen (see figure 5905D).

5.7 Calculations.

$$\text{Percent consumed} = \frac{L-A}{L} \times 100$$

Where: L = Original length of specimen.

A = Length of uncharred part of specimen from the top of the specimen down the side with less charred area to the point at which the uncharred area first reaches a width of less than 1 inch (25 mm).

5.7.1 To obtain the length of the uncharred portion, measure the uncharred length from the top of the specimen down the side of the specimen to the point at which the uncharred area first reaches a width of less than 1 inch (25 mm) from a side of the specimen.

5.7.2 Measure this length of the uncharred portion to the nearest 1/4 inch (1 mm) 1

6. REPORT

6.1 Report the test results for the 12-second flame exposure as follows:

The material:

- (a) ignites, propagates flame
- (b) ignites but is self-extinguishing
- (c) is ignition resistant
- (d) melts
- (e) shrinks away from the flame
- (f) drops flaming pieces.

METHOD 5905.1

6.2 The after-flame of the sample unit shall be the average of the results obtained of the individual specimens tested and shall be reported to the nearest 1 second.

6.3 For materials which melt or shrink away from the flame, report the after-flame only.

6.4 The percent consumed of the sample unit shall be the average of the individual specimens tested and shall be reported to the nearest 1 percent (see 5.7).

6.5 The individual results utilized in obtaining the average shall also be reported.

7. NOTES

7.1 Suggested sources of materials and equipment.

7.1.1 Gas mixture (4.6) and regulator valve system (4.7) are available from:

(a) Matheson Gas Products
P. O. Box 85
East Rutherford, NJ 07073

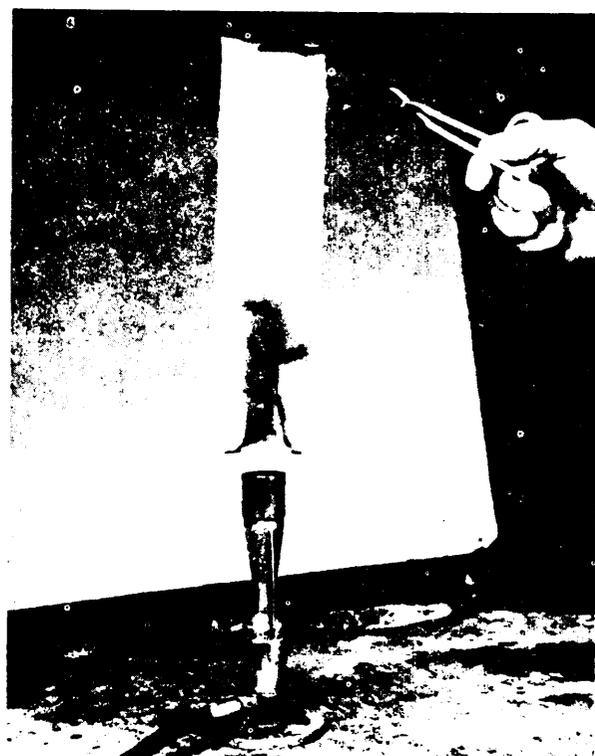
(b) Air Products and Chemicals, Inc.
P. O. Box 538
Allentown, PA 18105

7.1.2 Burner described in 4.1 is available from:

(a) Fisher Scientific Company
711 Forbes Avenue
Pittsburgh, PA 15219



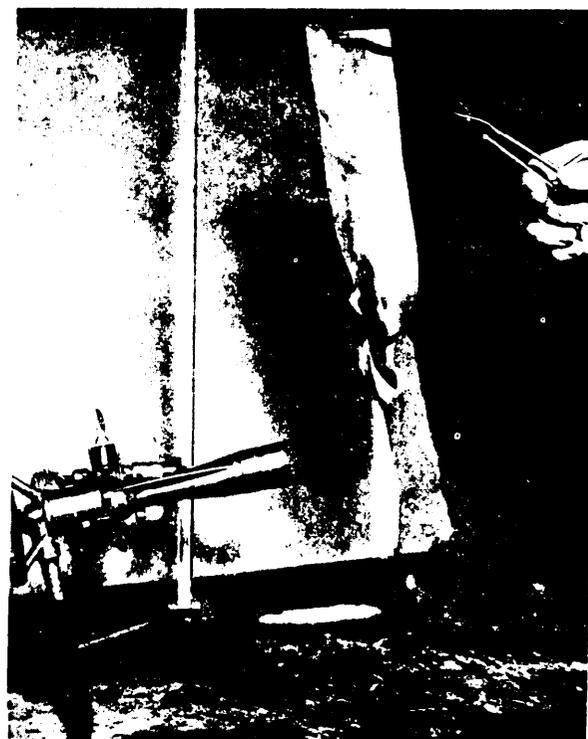
5905A



5905B



5905C



5905D

FIGURE 5905.1 Flame Contact Test

